\* \*DE 19713638-A1 BASE AG 97,04.02 97DE-1013638 (98,70,08) C08B 13/00, C08J 3/28, C09B 67/20, C09D 11/02, 17/00, C09K 19/38

Liquid crystal ester compounds for special-effect paint - comprise mixed ester(s) derived from hydroxyalkyl-cellulose ether and a mixture of saturated carboxylic acid and unsaturated mono; or di:carboxylic acid

C98-159908

Addnl. D.t.: KELLER H, MAXEIN G, MUELLER M, ZENTEL R

Liquid crystal (LC) or cholesteric mixed esters (I) derived from (a) hydroxyalkyl ethers of cellulose with (b) saturated aliphatic or aromatic carboxylic acids and (c) unsaturated mono- or di-carboxylic acids are new

Also claimed are

(i) LC or cholesteric polymers (LCP) obtained by radiation-hardening of di.

(ii) pigments containing LCP.

(iii) coating materials, especially paints, containing (I) or LCP containing pigments,

(iv) a process for the production of (I) by reacting a mixture of the acid

chlorides of (b) and (c) with component (a), and (v) a process for the production of pigments by pulverising LCP under mild thermal conditions, especially in an air-jet mill

<u>USE</u>

Esters (I) or polymers (LCP) or LCP containing pigments are used for the production of optical components or as coating materials, and esters (I) are used as colouring agents, especially as colour components of paint systems for coating surfaces or as components of printing ink (claimed). Applications include painting or coating cars, motorbikes, packaging, labels and ornamental articles.

<u>ADVANTAGE</u>

Esters (I) are one-component systems with better handling properties than esters of (a) and (c) alone (particularly with regard to spontaneous crosslinking), which act as thermotropic systems without the aid of solvents. This enables the economical production of LCP with good storage stability and with a precisely adjustable and uniform colour which can be permanently fixed to give a particularly DE 19713638-A+

impressive colour effect.

## PREFERRED COMPOSITION

The ether-linked hydroxyalkyl (OHA) groups in (a) comprise linear or branched 2 10C OHA groups, especially hydroxypropyl and/or hydroxyethyl.

The anhydro- glucose units in the cellulose are OHA-etherated with an average molar degree of substitution (DS) of 2-4, and component (a) has a molecular weight of 500-1000000.

Component (b) comprises linear 1-10C (preferably 2-6C) carboxylic acids, branched 4-10C (preferably 4-6C) carboxylic acids, or linear or branched halo- carboxylic acids, especially acetic, propionic, n- or iso- butyric, or n-valeric acid, preferably propionic, 3chloro- propionic, or n- or iso-butyric acid.

Component (c) comprises unsaturated 3-12C (especially  $\alpha.\beta$ ) unsaturated 3-6C) mono- or di-acids or their half-esters, preferably (meth)acrylic, crotonic, vinylacetic, maleic, fumaric or undecenoic acid, most preferably (meth)acrylic acid.

Component (a) is esterified by (b) and (c) with an average DS of 1.5-3, preferably 1.6-2.7, especially 2.3-2.6, and 1-30 (preferably 1-10, more preferably 5-7) % of the hydroxyl groups in (a) are esterified with component (c).

**EXAMPLE** 

5 g hydroxypropylcellulose (45 mmols functional groups, vacuum dried at 50°C) was dissolved in 60 ml acetone, treated with 270 mg acryloyl chloride and 0.5% phenothiazine, stirred for 1 hour at room temperature and refluxed for I hour. The cooled mixture was then treated with 12 ml propionyl chloride, stirred and refluxed as before, cooled, diluted with 40 ml acetone and worked up by precipitation in 800 ml ice-water followed by reprecipitation from acetone/ice-water and vacuum- drying at 40°C, to give 4.8 g mixed ester (I). A solution of 1 g (1) in a mixture of 10 ml acetone and 20 mg ethyl 2,4,6trimethylbenzoylphenylphosphinate (photoinitiator) was evaporated on a glass plate to give a thin film which showed selective reflection and could be hardened by irradiation with UV. A layer of the evaporated material between two glass plates was irradiated after. hour with UV. A light to give a polymerised film with selective reflection at a max, wavelength of 570 nm; this film appeared green when viewed directly and blue when seen from the side. The selective reflection of such films was dependent on the temperature during the UV-curing process. (HW) (5pp1712DwgNo.0/0)

DE 19713638-A